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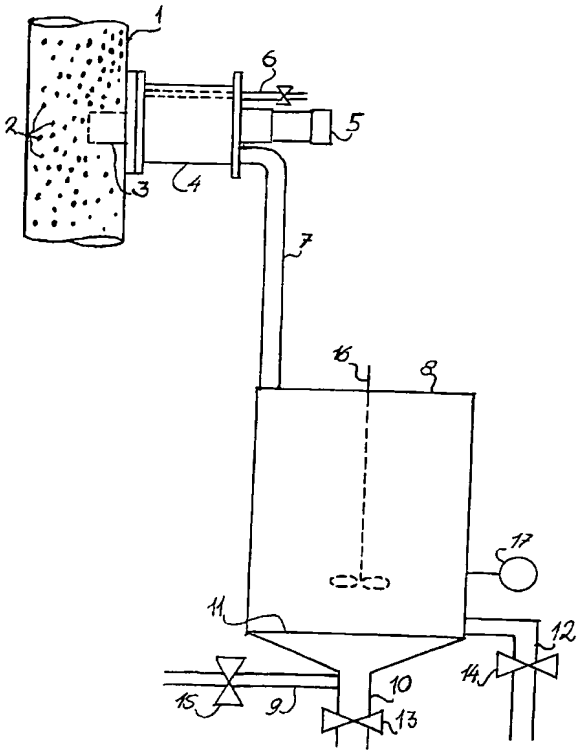
(54) Title: METHOD AND SYSTEM OF APPARATUS FOR EXTRACTING LIQUID FROM CELLULOSE PULP OF HIGH PULP CONSISTENCY FOR ANALYSIS PURPOSES



(57) Abstract: The present invention relates to a method and to a system of apparatus for automated, intermittent extraction of liquid from liquid containing cellulose pulp of high pulp consistency for analysis of the chemical content of said liquid, wherein a collecting unit of given volume is inserted into a transport line or a vessel, wherein the cellulose pulp is advanced in a solid, voluminous state and the collecting unit, together with its content of cellulose pulp, is withdrawn from the transport line or the vessel and moved into a storage space located in connection with the transport line or vessel, and wherein the cellulose pulp is caused to leave the collecting unit and the cellulose pulp is then transported further in an unchanged or lowered pulp consistency, characterized in that the cellulose pulp is delivered to a dilution space to which there is supplied a controlled amount of water together with the cellulose pulp and/or explicity so as to obtain a pulp fibre suspension of comparatively low pulp consistency; and in that the pulp fibre suspension is thereafter de-watered so as to obtain a pulp fibre cake and a flow of liquid that is essentially fibre-free, wherein said liquid flow is removed from the system and passed to an analysis position, whereafter the dilution space is freed from pulp fibres so as to prepare the system for the extraction of fresh liquid for analysis.

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Method and system of apparatus for extracting liquid from cellulose pulp of high pulp consistency for analysis purposes

Technical field

The present invention relates to a method and to a system of apparatus for extracting liquid from cellulose pulp of high pulp consistency for the purpose of analysis. By cellulose pulp is meant any known cellulose pulp whatsoever produced from any lignocellulosic material whatsoever. Cellulose pulps can be divided into the following groups, chemical pulp, semichemical pulp and mechanical pulp in respect of their yield, i.e. how much of the starting material that is obtained in the form of pulp and in a yield scale ranging from low to high. These groups can, in turn, be finely graded. For example, chemithermomechanical pulp (CTMP) thermomechanical pulp (TMP) and groundwood pulp (GWP) fall within the group designated mechanical pulps, due to the fact that all of said pulps have a yield that exceeds 90%.

Many process stages are employed in the manufacture of such pulps, and then particularly in the manufacture of bleached pulps. In these various process stages, during which different chemicals are often used, the pulp manufacturer is interested in the ability to take samples of the process liquid (also referred to as filtrate), preferably in an automated fashion, so as to be able to conduct different liquid analyses. For instance, the manufacturer will normally wish to analyse the liquid to determine how much of a given bleaching agent that remains in the liquid phase after the bleaching stage, i.e. the amount of so-called residual bleaching agent present.

The need to extract liquid from cellulose pulp of high pulp consistency for analysis purposes is also found in the manufacture of waste paper pulp and in paper mills. It is not unusual in paper mills to treat the pulp, for instance to bleach the pulp, in one or possibly more stages in an introductory phase, i.e. at some point prior to stock preparation. A typical bleaching agent in this connection is some form of peroxide.

Background art

The pulp consistency of the pulp suspension, which can vary from process stage to process stage during the pulp manufacturing process, is of decisive significance with regard to the difficulty in taking a relevant liquid sample from a pulp suspension. Expressed simply, it can be said that the lower that pulp consistency the easier it is to take a relevant liquid sample, and it is irrefutable that it is extremely difficult to take a relevant liquid sample or filtrate sample at very high pulp consistencies, for instance at consistencies ranging from 20% and upwards.

Several filtrate-sampling devices are described in the literature, including the patent literature.

U.S. Patent Specification 5,625,157 teaches such a sampler whose front end is inserted into a vessel or container that contains a pulp suspension. The sampling device comprises a partially hollow cylinder that includes a centrally positioned rod, which can be moved both forwards and backwards. One end of the rod merges into a solid cylindrical body which extends within and at a given short distance from the inner wall of the front end of the sampling device, which is open at said end and in direct contact with the pulp suspension. When the rod is fully withdrawn, the solid body rests against a seating, which seals and prevents suspension liquid from penetrating beyond the solid body and into a cavity connected to an outlet or drain. When the solid body is moved to an intermediate position by means of the rod, the suspension liquid is free to flow into the space between the solid body and the inner cylinder wall and past the seating and into said cavity and from there to an analyser, for instance, via said outlet. Pulp fibres are collected furthest up in the cylinder during this process, so as to form a fibre cushion or mat through which the liquid or the filtrate can flow. This cushion acts as a filter which retards approaching pulp fibres and allows fibre-free filtrate to pass through. When sampling of the filtrate has been completed, the solid body is moved by the rod to its end position, edge-to-edge with the

outer end of the cylinder, therewith causing the resultant fibre cushion to be pressed into the pulp suspension and mixed therewith. The sampling device can be back-flushed with water, i.e. cleaned, when so desired.

The descriptive part of the patent specification is very concise and no mention is made as to how the suspension liquid or the filtrate is driven from the pulp suspension container and through the sampling device. It is understandable that what has been described will take place when an overpressure prevails in the pulp suspension container. If no overpressure prevails in the pulp suspension container, it is probable that the filtrate must be withdrawn by suction. No mention is made as to how high up in the pulp consistency scale the sampling device functions. The device probably functions at a medium consistency at the highest, i.e. at a highest consistency of perhaps 12%.

Swedish Patent Specification 511 069 (9802526-5) discloses a device for taking a filtrate sample from a pulp suspension. The device includes a piston rod which can be moved forwards and backwards within a lining by means of a cylinder and which includes a filter that is intended to capture a sample in an outer position. The device is characterised in that the filter is comprised of a generally three-edged thread, forming thread elements which are disposed tightly adjacent each other with their base surfaces directed outwards and their converging surfaces directed inwards towards the centre of the filter, and wherein the filter can be cleaned during its backward movement into the lining, by scraping from the lining those fibres that have fastened to the filter. This specification also fails to disclose the pulp consistencies at which the liquid sampling device functions. It is likely also in this case, that the device functions at a medium consistency at the highest, i.e. a consistency of perhaps 12% at the highest.

There is described on pages 10 and 11 of an article written by B.S. Terekin and L.N. Toropov published in the Russian journal Tsell Bum Karton Ref. Inform, No. 16 with the heading (translated first into Swedish and then into English) "Equipment for Extracting Liquid from Cellulose Pulp of High Consistency". Also described is a liquid sampling device. The authors of the article have said that by high pulp consistency is meant a consistency of 10 to 14%.

In this instance, the actual sample-taking device has the form of a perforated pipe stub inserted into a pulp suspension line. The pipe stub is embraced by a wire cloth. A pressure of 0.1-0.3 MPa prevails in the pulp suspension line. Liquid in the pulp suspension

is forced through the wire cloth and the perforations in the pipe stub and from there to and through a switch valve. The liquid is pressed from the valve to a collecting vessel in which air is separated from the liquid. Air-free suspension liquid or filtrate is passed from the collecting vessel to an assaying or analysing cell. The switch valve is a particular feature in this context. Pulp fibres collect on the wire cloth as sample liquid is taken from the pulp suspension. Sampling of liquid is stopped when the pulp fibre mat has grown to a given thickness, and compressed air is, instead, blown back to the perforated pipe stub and to its embracing wire cloth, via the switch valve. The wire cloth is thereby freed from the pulp fibre mat and sampling of liquid from the pulp suspension can be continued upon completion of this operation.

There is nothing suggested in this article that the sample-taking device concerned is able to function at pulp consistencies above 14%. However, high consistency bleaching of cellulose pulp, for instance, has long been known to the art. A pulp consistency of 25-30% is often concerned in the oxygen gas bleaching of pulp. Still higher pulp consistencies, such as consistencies up to 35%, sometimes exist in the case of the peroxide bleaching of cellulose pulp, for instance mechanical pulp. These very high pulp consistencies can also exist when using other bleaching agents, for example chlorine dioxide.

A major interest in all of these bleaching processes and not least in a peroxide bleaching process, is the knowledge of how much of the bleaching agent charged remains after the bleaching process. A primary interest in this regard is to be able to control the bleaching process optimally. It may also be of value to know, for instance, the residual peroxide content of a bleaching waste liquor when it is desired to charge the cellulose pulp with this chemical-containing liquid in some other position in the pulp manufacturing chain. Commercially available functional analysis methods and analysis apparatus for determining residual peroxide, for instance, have long been known to the art. However, these known methods fail with regard to the extraction of sample liquid. Because the pulp suspension is already pressed very hard, one difficulty resides in the fact that it is difficult to squeeze or press further liquid from the pulp suspension. Even though a small amount of liquid or filtrate can be successfully pressed from the pulp suspension, the amount obtained is so limited as to be insufficient for analysis. In order to be successful in sufficient filtrate

for analysis in accordance with the press method, it is necessary to take out and/or treat an unrealistically large quantity of pulp suspension.

Disclosure of the invention

Technical problem

As will be apparent from the aforegoing, in respect of the manufacture of unbleached and bleached cellulose pulp and in the manufacture of waste paper pulp and in paper mills, there is a need to obtain an appropriate quantity of process liquid or filtrate in order to analyse the filtrate, for example in order to determine the concentration of a given chemical prior to, within, or after certain process stages that are performed at high pulp consistencies. This recovery will preferably take place in an automated and operationally reliable manner that minimises the requirement of operator supervision.

The solution

This need is satisfied and the problems solved by means of the present invention which relates to a method for automated intermittent extraction of liquid from liquid-containing cellulose pulp of high pulp consistency for analysis of the chemical content of said liquid, wherein the method comprises inserting a collecting unit of given volume into a transport line or a vessel in which the cellulose pulp is advanced in a solid, voluminous state, wherein the collecting unit and its content in the form of a cellulose pulp sample are withdrawn from the transport line or said vessel and moved into a storage space in connection with said transport line or vessel wherein the cellulose pulp sample is removed from the collecting unit, and wherein the cellulose pulp sample is then further transported at an unchanged or lowered pulp consistency. The method is characterised by introducing the cellulose pulp sample into a dilution space to which a controlled amount of water is added together with the cellulose pulp sample and/or explicitly so as to obtain a comparatively low pulp consistency in the pulp fibre suspension; and thereafter dewatering the pulp fibre suspension so as to obtain a pulp fibre cake and an essentially fibrefree liquid flow, said liquid flow being removed from the system and passed to an analysis position, whereafter the dilution space is freed from the pulp fibres and the system restored for extraction of fresh liquid for analysis.

With regard to the determination of high pulp consistency, there is no distinct lower limit and of all accepted lowest percentage. It can be said that the lower limit lies somewhere within the consistency range of 10-15%, and that all consistencies thereabove are included in said definition. Pulp consistencies in excess of 40% are unusual in practice. It can be mentioned by way of example that high consistency peroxide bleaching is normally carried out within the pulp consistency interval of 30-35%.

The removal of liquid-containing cellulose pulp in a solid, voluminous state can, in principle, be effected in any position whatsoever, although from the aspect of sampling uniformity it is highly preferred that the cellulose pulp is generally finely-divided and in some form of movement. It is highly suitable to take the pulp sample by inserting the collecting unit into a pulp transporting line that has the form of a chute or downfeeder located in connection with and beneath the end of a pulp screw feeder, where the solid, voluminous and, to some extent, finely-divided pulp falls freely into the chute.

The collecting unit, which can also be referred to as a ladle, may have a semi-cylindrical shape with the open side facing upwards, so that some of the downwardly falling tiny pieces of cellulose pulp, or pulp fluff, land in the ladle. When the ladle is full, it is withdrawn from the chute and moved into a storage space, for instance by means of a rod and, for instance, pneumatically. The storage space is located adjacent the chute and the wall of the chute includes an opening that has the same shape and the same area as the cross-sectional shape and cross-sectional area of the ladle. This means that any surplus pulp heaped on the ladle will be scraped off from the ladle as it is drawn into the storage space. This means that each cellulose pulp sample that is taken will have essentially the same volume. (It is possible that the extent to which the pulp pieces are packed in the ladle will vary somewhat). Provided that the fluffiness of the pieces of pulp in the chute is essentially the same over the passage of time, and provided that the packing degree of said pulp pieces is essentially the same on each sampling occasion, the amount of pulp sampled will also be essentially the same on each occasion. Assuming that 100 grams of pulp is removed on each sampling occasion and that the pulp consistency is 30%, this will mean that the storage space will contain 30 grams of pulp fibres and 70 grams of chemical-containing suspension liquid or filtrates.

The pulp sample taken must be moved from the ladle down into the lower part of the storage space. This can be effected in several ways. One way is to turn the ladle

upside down. If some of the pulp still remains in the ladle, this residue can be removed with the aid of jet of compressed air. Alternatively, there can be used a perforated ladle onto which water is sprayed so that the pulp fibres will accompany the water down through the ladle perforations. This spray water must be clean and the amount of water used in this connection must be measured.

As will be apparent from the aforegoing, the amount of pulp removed on each sampling occasion is based on the volume of the described collecting unit combined with the fluffiness of the collected pulp pieces and the packing degree of said pieces. Should there be variations in any of the variable parameters, this will be apparent by virtue of the fact that, for instance, the aforesaid 70 grams of suspension liquid will not be obtained in the storage space on each sampling occasion. A variation in plus or minus one or a few grams with respect to the amount of suspension liquid can be tolerated, in view of the limited error in the analysis result caused by such variation. These possible variations are neutralised, by providing the collecting unit or the storage space with a sensor that determines and indicates the true weight of the pulp sample.

The pulp sample, with unchanged or lowered pulp consistency, is passed to a dilution space, for instance in the form of a tank, such as a cylindrical container. A controlled amount of dilution water is normally also fed to the tank or container. This dilution water and also the water that may be delivered to the storage space must be clean, and may consist of chemically cleaned and/or ion-exchanged water. The amount of dilution water added is determined by the pulp consistency desired in the diluted pulp suspension. This consistency is suitably 0.5-3%. The pulp fibres are agitated or stirred to some extent as the dilution water is added in the container. The manner and degree of this agitation is determined to some extent by the manner in which the conduit, through which the dilution water is normally delivered, is connected to the container, for instance whether the conduit is connected tangentially or connected directly or indirectly to the bottom part of the container. If the agitation caused by the supply of dilution water is insufficient, the container is equipped with an agitator or stirrer of any known kind, for instance a propeller agitator.

A pulp consistency gauge can be connected to the dilution space, for instance in the form of the aforesaid container, so as to know precisely the amount of suspension

liquid that has been extracted via the cellulose pulp sample. Such a gauge functions effectively at the aforesaid pulp consistency range.

The bottom part of the dilution space, for instance the bottom part of the cylindrical container, includes a de-watering device by means of which the pulp fibres and the diluted suspension liquid are mutually separated. The diluted suspension liquid is removed from the dilution space and passed to an analysis position, which includes, for instance, a collection vessel in which the amount of liquid delivered is measured, suitably in a given number of millilitres.

The liquid is then analysed as required. If a sample is taken of cellulose pulp that has just been bleached with peroxide, for instance, the residual peroxide content is determined in grams per litre, for example. When the amount of suspension liquid that had a given peroxide content that accompanied the pulp sample, for instance 70 grams in accordance with the aforesaid, is known and the amount of dilution water added to the system on one or two occasions is also known, it is possible to calculate from the measurement result of the diluted suspension liquid the residual peroxide concentration in, e.g., grams per litre of the suspension liquid present in the pulp after the peroxide bleaching stage.

It is necessary to free the dilution space of its pulp fibre content before taking a fresh cellulose pulp sample. This can be achieved by back-flushing with water, so as to slush the pulp fibre cake present and convert the same to a fibre-sparse pulp suspension which leaves the dilution space via a separate outlet or drain. These pulp fibres in the form of a pulp suspension can be recovered and admixed with the main pulp suspension.

The present invention also relates to a system of apparatus for automated, intermittent extraction of liquid for the analysis of its chemical content from liquid-containing cellulose pulp of high pulp consistency, including a reciprocatingly movable collection unit of given volume which is inserted into a transport line or a vessel in one position, where the cellulose pulp is advanced in a solid, voluminous state, and is withdrawn from the transport line or the vessel together with its content in the form of a cellulose pulp sample in a second position and passed into a storage space located in connection with the transport line or vessel, said storage space temporarily housing the cellulose pulp sample and includes an outlet that has a conduit connected thereto. The system is characterised in that the conduit is connected to a dilution space of adapted

volume, wherein said dilution space includes means for de-watering the pulp fibre suspension resulting from dilution of the cellulose pulp with water so as to produce a pulp fibre cake and a substantially fibre-free liquid flow; and in that a further number of conduits are connected to the dilution space, such as a conduit for removing the essentially fibre-free liquid flow from the system and for transportation of said liquid to an analysis position, and one further conduit for removing the pulp fibre cake in a slurried state from the system, and one further conduit for supplying dilution water, wherein this latter conduit may be connected to the first mentioned conduit or connected in the conduit for removing the essentially fibre-free liquid flow or may be detached and/or connected to the storage space, and one further conduit for delivering backflushing liquid, wherein this latter conduit may be connected to the conduit for removing said essentially fibre-free liquid flow or may be detached.

The means for dewatering the pulp suspension obtained in the dilution space may have a number of different constructions. The lowermost part may be covered with a porous material, optionally obtained by sintering, which retards the pulp fibres and allows the suspension liquid to pass through. Porous glass materials are available. Another typical material for retarding and capturing pulp fibres and allowing liquid to pass through is wire cloth. Wire cloths of varying mesh sizes are available. The mesh size is normally given with a mesh number that indicates the number of threads disposed at a given distance apart per inch, i.e. per 25,4 millimetres. The higher the mesh number, the finer the mesh of the wire cloth. The wire cloth may be placed against or fastened to the wall or walls of the dilution space. The wire cloth may also rest on a perforated substrate, which is anchored in or at the dilution space.

Certain of the aforesaid conduits include openable and closeable valves. As before mentioned, it is necessary to measure the amount of dilution liquid supplied to the pulp sample. If it is decided that precisely the same amount of dilution fluid shall be delivered to the pulp sample on each occasion, the conduit through which the dilution liquid, i.e. clean water, is delivered need not include a valve. However, several advantages are obtained when this conduit is fitted with a valve. There is often available a conduit system in which the water is under a given pressure. When a dilution liquid conduit having, for instance, a magnetic valve is connected to such a conduit system, precisely the amount of dilution liquid to be delivered can be determined by causing the magnet to hold

the valve open for a given length of time. The conduit through which suspension liquid is removed from the dilution space will preferably include a valve that is held closed during the dilution process and also during a possibly following pulp consistency measuring process. Similarly, a detached back-flushing conduit will preferably include a valve, which also applies to the conduit through which the consumed pulp fibres are flushed out.

Advantages

The invention enables, for instance, the residual bleaching agent content of a suspension liquid to be analysed in any position desired by the skilled person, for instance immediately after a high consistency bleaching stage. The skilled person has earlier been forced to carry out the analysis, if it has been carried out at all, at a certain distance from the bleaching stage where the suspension liquid has undergone a change, for instance by being mixed with other chemical containing or chemical consuming liquids.

Another advantage is that the system of apparatus is robust and includes only a few and essentially only one movable part, namely the collecting unit, meaning that the system is highly reliable in operation.

Description of the drawing

Figure 1 of the accompanying drawing illustrates schematically an inventive system of apparatus and also an application of the inventive method.

Best embodiment

There is described in the following with reference to Figure 1 an application of the inventive method and an inventive apparatus system. At the same time, certain stages or steps are described in more detail than was earlier the case. There is finally described a test that has a bearing on the inventive method to some extent.

Pulp in a solid, voluminous state falls down gravitationally through the vertical pipe 1, which may be a chute, located at one end of a pulp screw feeder. It can be asserted that a very large number of tiny pieces of pulp 2, or fluff, will fall down the pipe. Some of these tiny pulp pieces 2 of high pulp consistency will land in an upwardly open ladle 3, which is inserted slightly and temporarily into the pipe 1 through a hole in the pipe wall. The ladle 3 may have any appropriate shape, for example a semi-cylindrical shape.

When a desired quantity of pulp pieces have been captured, for instance when the semi-cylindrical ladle is filled to the brim or said pieces are heaped on the ladle, the ladle 3 is withdrawn from the pipe 1 and moved into the storage space 4. The ladle 3 can be moved between the two described positions pneumatically and is driven through a compressed air cylinder device 5. In this case, the ladle 3 is attached to a rod whose opposite end is fastened in the cylinder. When compressed air is delivered to the left end of the device 5, the ladle 3 is withdrawn from the pipe 1, whereas the ladle 3 is pushed into the pipe 1 when compressed air is applied to the right end of said device 5. The hole provided in the pipe wall will preferably have the same shape and size as the size and shape of the ladle cross-section, for instance a semi-circular shape. Any pulp that is heaped on the ladle will be scraped off as the ladle 3 is withdrawn into the storage space 4, which may be cylindrical.

In the next stage, the pulp sample is moved from the ladle 3 to the lower part of the storage space 4. This can be achieved in several ways. The ladle 3 may be provided with perforations, such as round holes or elongate slots, through which the pulp fibres are transported with the aid of clean water sprayed over the pulp sample and delivered via the conduit 6. The amount of water delivered must at least be sufficient to remove all pulp fibres from the ladle and, thereafter, also from the storage space 4 and down through the conduit 7 to the dilution space 8 or, if desired, to a dilution tank of any configuration whatsoever, for instance a cylindrical or parallelepipedic shape. As a result of supplying dilution water via the conduit 6, the pulp consistency of the pulp sample is considerably lowered already in the storage space 4.

The pulp consistency of the pulp sample in the tank 8 is lowered still further, by supplying dilution water through the conduit 9, said conduit being connected to the conduit 10. A wire cloth 11 is applied in the bottom of the tank 8. The mesh number is about 40. The tank 8 also includes an outlet in the form of the conduit 12. The valve 13 in the conduit 10 and the valve 14 in the conduit 12 are closed before delivering dilution water through the conduit 9. Because dilution water is delivered from beneath and up through the wire 11 and into the tank 8, the pulp fibres are spread roughly uniformly throughout the whole of the tank 8 above the wire 11. As a result, the subsequent de-watering stage is effected in an optimal manner.

In the dewatering stage, the valve 14 is still kept closed and, at the same time, the valve 15 that controls the delivery of dilution water is closed and the valve 13

opened. The pulp fibres therewith collect to an increasing extent on the wire 11, so as to form a pulp fibre bed or cake, at the same time as the suspension liquid flows through the conduit 10 to a liquid collecting position, whereafter all of the liquid or a determined part thereof is analysed, for instance with respect to its bleaching agent content.

Distribution of the pulp fibres evenly over the full volume of the tank 8 above the wire 11 is facilitated when using some form of agitator or stirrer, for instance a propeller agitator 16.

As before mentioned, by using a pulp consistency measuring device or gauge 17 it is possible to achieve practical full control of how many grams of bone dry pulp fibres are included in the pulp in the pulp sample taken from the system and how many grams of suspension liquid or filtrate are included in said pulp sample. In this regard, it is very important to check and measure the total quantity of dilution water added, as is normal. Many different types of consistency gauges are available. One group is based on shear force measurement, such as rotating shear force gauges or shear force gauges equipped with active blades or shear force gauges equipped with static blades. Another group is optically based, such as optical measurement by transillumination and optical measuring by light reflection. Also available are gauges based on microwave measurement. At least one of these consistency gauges can be used appropriately in the present context. In order to obtain a correct measuring result of the pulp consistency, it is preferred to use additional agitation during the measuring process, for instance with the aid of the propeller agitator or stirrer 16.

The extent to which the pulp sample is diluted, i.e. the consistency of the pulp suspension in the dilution tank 8 immediately prior to the dewatering stage, is dependent on several factors. One factor is the pulp consistency of the pulp sample taken. Another factor is the amount of liquid desired for carrying out the analysis. A third factor is the content, e.g. of residual bleaching agent in the pulp sample taken. The amount of dilution liquid added, i.e. clean water, must not be so high that the content of the bleaching agent in the resultant aqueous solution will be so low as to prevent it from being analysed or make analysis difficult to carry out. As before mentioned, the pulp consistency will, in the majority of cases, suitably lie somewhere within the range of 0.5% to 3%.

In order to be able to reuse the pulp sampling system and the filtrate removal system coupled thereto, it is necessary to remove the pulp fibre bed formed on the wire 11.

This can be achieved by keeping the valve 13 closed and the valve 14 open as flushing liquid, in the form of water, is delivered through the conduit 9. The pulp fibres therewith leave the wire 11 and convert to a pulp fibre suspension of very low pulp consistency, wherewith the suspension leaves the dilution tank 8 through the conduit 12. These pulp fibres can be recovered and returned somewhere in, e.g., the pulp manufacturing system or to the stock in a paper mill. However, the amount of pulp fibres recovered is so small as not to constitute any direct material loss if the pulp fibres are allowed to exit the system.

With respect to the distribution of the quantity of dilution liquid delivered via the conduit 6 and via the conduit 9, it is fully possible to deliver the total amount of dilution liquid through the conduit 6, meaning that the conduit 9 can be omitted. In this case, the conduit 10 can be used as such for back-flushing of the system. However, such dilution liquid supply is not preferred, since problems may occur in obtaining uniform distribution of the pulp fibres throughout the whole of the dilution tank 8 above the wire 11 immediately prior to the dewatering stage, including a possible pulp consistency measuring process. In such a case, the operator is highly dependent on well-functioning additional agitation or stirring, for instance by means of the propeller agitator 16. It is preferred to deliver at least some of the dilution liquid, i.e. clean water, directly to the dilution tank 8 and, e.g. from beneath, through the conduit 9, as shown in Figure 1.

In the case of another embodiment of the ladle 3 and the storage space 4, the supply of dilution liquid via the conduit 6 is unnecessary. The ladle 3 may be devoid of perforations and the semi-cylinder may, for instance, comprise two mutually connected halves which are folded upwards and away from each other within the storage space 4, so that the pulp sample will fall gravitationally onto the bottom of the storage space 4. The pulp sample need not be taken from the space 4 via said wall, as in the case of the Figure 1 embodiment. Alternatively, the lower part of the storage space 4 may be given a funnelshaped configuration and connected to a conduit 7 like the one shown in Figure 1. In this case, the pulp sample moves from the ladle 3 all the way to the dilution tank 8 exclusively under the effect of gravitation. If, against expectations, not all of the pulp fibres leave the ladle 3 and/or the storage space 4, these remaining fibres can be removed with the aid of compressed air. In this case, it is necessary to connect a dilution liquid conduit directly or indirectly to the dilution tank 8, in accordance with one of the aforedescribed methods.

A small amount of dilution liquid can also be delivered to the storage space 4 in the case of the newly described embodiment of the invention. For example, the cleaning medium that may be required can consist of water instead of compressed air.

The ladle 3 need not be perforated nor divided, but may be emptied of its pulp sample content by turning the ladle upside down in accordance with what had been earlier described. It will be understood from this that a large number of structural designs have been described and that other designs compatible with the system lie within the concept of the invention.

The aforedescribed method can readily be automated. It has long been known that valves can be remotely controlled. Moreover, the insertion and withdrawal of the ladle 3 into and out of the pip 1 can also be remotely controlled with no difficulty. The time lapse between taking pulp samples is determined, downwardly calculated, by the time taken to carry out the described cycle of events and, in other respects, on how quickly the filtrate can be analysed and on the number of analyses desired, e.g., per hour in the individual case. All of the described measures can be computer controlled, meaning that the work required by the operator is confined to entering the desired measures and parameters into his computer, coupled with a certain amount of supervision.

Example 1

In a pulp mill for producing thermomechanical pulp from spruce wood bleached in one bleaching stage with hydrogen peroxide to a brightness of about 68% ISO, samples of the bleached pulp were taken manually from the bottom of a bleaching tower. The pulp consistency during the bleaching stage was 31.6%. The amount of hydrogen peroxide added corresponded to 9.5 kg per tonne of absolute dry pulp. Sodium hydroxide and a number of additive chemicals were added to the pulp immediately upstream of the bleaching tower, in a mixer.

400 millilitres of this pulp in flake form was introduced into a potato press, i.e. a kitchen implement designed to produce pressed potato, wherewith the pulp was pressed manually in said instrument to an extent at which the operator managed to recover 20 grams of liquid or filtrate. The manner in which the filtrate was analysed with respect to its hydrogen peroxide content is described below.

Another part of this pulp was introduced into a container and the pulp was then fluffed to some extent with the aid of an electrically operated whisk, which was also in the form of a cooking implement. This was done so that the pulp would obtain roughly the same state of agglomeration as the pulp in certain positions in a pulp mill, for instance in a chute located beneath the end of a pulp feeding screw. It was found that the volume of 100 grams of said pulp was 470 ml. 170 grams of this pulp was passed to a container together with 2300 grams of chemically cleaned water. The mixture was agitated for a short period of time with the aforesaid electric whisk, wherewith the resultant pulp suspension had a pulp consistency of 2.2%. The pulp suspension was then poured into a funnel-like container provided with a wire cloth at its lower part. The wire cloth had a mesh number of 40. As a result of the ensuing autopressure, a pulp fibre cake was obtained on the wire cloth and a fibre-free flow of liquid, corresponding to more than 2 litres, left the funnel and was captured in a container. This liquid was analysed with respect to its hydrogen peroxide content in accordance with the following description.

The analysis was not carried out industrially, but in a laboratory, wherein, e.g., 3 grams of filtrate were passed into a beaker (the exact weight was noted) together with 60 millilitres of distilled water. There was then added 2 ml sulphuric acid (10%), 2 ml potassium iodide (0.3 M) and 3 drops of ammonium molybdate (10%). The resultant solution was stirred for a period of some minutes. The sample, or solution, was then titrated with 0.1 N sodium thiosulfate while using a redox electrode as a detector.

The added potassium iodide reacted with the hydrogen peroxide present, so as to form free iodine (I_2). During the titration process, the iodine reacted with the added thiosulfate in accordance with the formula

$$2\ S_2O_3^{2-} + I_2 \rightarrow S_4O_6^{2-} + 2I^-$$

The amount of hydrogen peroxide present in the filtrate was calculated in the following manner and with the aid of the following formula:

$$\text{Hydrogen peroxide or residual hydrogen peroxide in g/l} = \frac{X \cdot 0.1 \cdot 34}{2 \cdot Y}$$ where

X = ml sodium thiosuslfate

Y = sample weight in grams and

34 = the molecular weight of hydrogen peroxide.

A single test was carried out on the filtrate (No.1) pressed from the pulp, while a double test was carried out on the diluted filtrate (No.2).

The results can be seen from the following table.

Table 1

Sample No.	Pulp Consistency	Residual Hydrogen Peroxide	
	%	g/l	kg/tonne pulp
1	31.6	1.79	3.9
2a	2.2	0.091	4.0
2b	2.2	0.088	3.9

The analysis results for both filtrates are the same and show that of the 9.5 kg hydrogen peroxide added per tonne of pulp, 3.9 kg per tonne of pulp remained after the bleaching stage.

Although sampling of the pulp, including the desired filtrate for tests 2a and 2b, was not carried out strictly in accordance with the invention, the example shows that dilution of the pulp sample taken with chemically cleaned water does not jeopardise the reliability of the analysis.

It is important to note that the amount of filtrate necessary for analysing, e.g. the amount of residual hydrogen peroxide industrially, i.e. in direct connection with pulp manufacture and out in the pulp mill, is much greater than the amount of filtrate required for laboratory analysis.

The amount of filtrate required for industrial analysis can approach one litre and if 20 grams or millilitres of filtrate can be successfully pressed from 400 millilitres of pulp in flake form, it is necessary to remove $50 \cdot 0.4 = 20$ litres of pulp in order to remove one litre of filtrate. A pulp quantity of this magnitude is both difficult to remove from the system and difficult to handle.

CLAIMS

1. A method for automated, intermittent extraction of liquid from liquid containing cellulose pulp of high pulp consistency for analysis of the chemical content of said liquid, wherein a collecting unit of given volume is inserted into a transport line or a vessel, wherein the cellulose pulp is advanced in a solid, voluminous state and the collecting unit, together with its content of cellulose pulp sample, is withdrawn from the transport line or the vessel and moved into a storage space located in connection with the transport line or vessel, and wherein the cellulose pulp sample is caused to leave the collecting unit and the cellulose pulp sample is then transported further in an unchanged or lowered pulp consistency, **characterized** in that the cellulose pulp sample is delivered to a dilution space to which there is supplied a controlled amount of water together with the cellulose pulp sample and/or explicitly so as to obtain a pulp fibre suspension of comparatively low pulp consistency; and in that the pulp fibre suspension is thereafter dewatered so as to obtain a pulp fibre cake and a flow of liquid that is essentially fibre-free, wherein said liquid flow is removed from the system and passed to an analysis position, whereafter the dilution space is freed from pulp fibres so as to prepare the system for the extraction of fresh liquid for analysis.

2. A method according to Claim 1, **characterized** in that the collection unit is inserted into a transport line in the form of a chute located in connection with and beneath the end of a pulp feeding screw, wherein the solid, voluminous and to some extent finely divided pulp falls freely into said chute therewith enabling representative pulp samples to be taken time after time.

3. A method according to Claims 1-2, **characterized** in that the collection unit or the storage space includes a sensor that indicates the weight of the cellulose pulp sample.

4. A method according to Claims 1-3, **characterized** in that at least the dilution process in the dilution space is carried out with the use of additional agitation or stirring.

5. A method according to Claims 1-4, **characterized** in that in that subsequent to having supplied all dilution water, the pulp consistency is measured in the dilution space by means of a connected pulp consistency measuring device while applying additional agitation or stirring.

6. A method according to Claims 1-5, **characterized** in that the dilution water added is clean water, such as chemically cleaned or ion-exchanged water.

7. A method according to Claims 1-6, **characterized** in that the pulp fibre cake is caused to form on a wire cloth of given mesh size disposed in the bottom part of the dilution space simultaneously with the chemical-containing liquid flows through said fibre cake concurrently with its formation, whereafter the chemical-containing liquid passes through the wire cloth and out of the system, via a liquid outlet, and up to an analysis position.

8. A method according to Claim 7, **characterized** in that subsequent to the liquid extraction the dilution space is back-flushed with water so as to slurry the pulp fibre cake and therewith convert the suspension to a fibre-sparse suspension that leaves the system via a separate outlet located above the wire cloth.

9. A system of apparatus for automated, intermittent extraction of liquid from a liquid containing cellulose pulp of high pulp consistency for the purpose of analysing the chemical content of said liquid, said system including a reciprocatingly movable sample collection unit (3) of given volume which in one position is inserted in a transport line (1) or a vessel where the cellulose pulp (2) is advanced in a solid, voluminous state, and which in another position is withdrawn together with its contents in the form of a sample of cellulose pulp taken from the transport line (1) or the vessel and moved into a storage space (4) located in connection with the transport line (1) or the vessel, said storage space (4) temporarily housing the cellulose pulp sample and including an outlet to which a conduit (7) is connected, **characterized** in that said conduit (7) is connected to a dilution space (8) of adapted volume, wherein said dilution space (8) includes means (11) for dewatering the pulp fibre suspension obtained after diluting said suspension with water,

said dewatering process resulting in a pulp fibre cake and a flow of liquid that is essentially fibre-fee; and in that a number of further conduits are connected to the dilution space (8), such as one conduit (10) for removal of the essentially fibre-free liquid flow from the system and for transportation of said liquid flow to an analysis position, and one conduit (12) for removing the pulp fibre cake in a slurried state from the system, and one dilution water supply conduit (9) which may be connected to the first mentioned conduit (7) or connected to the conduit (10) for the removal of the essentially fibre-free liquid flow or may be detached and/or connected (6) to the storage space (4) and one back-flushing liquid supply conduit (9) which may be connected to the conduit (10) for removing the essentially fibre-free liquid flow or may be detached.

10. A system of apparatus according to Claim 9, **characterized** in that the storage space (4) includes means for ensuring that all of the pulp sample taken will be removed from the collecting unit and arrive in the lower half of the storage space (4).

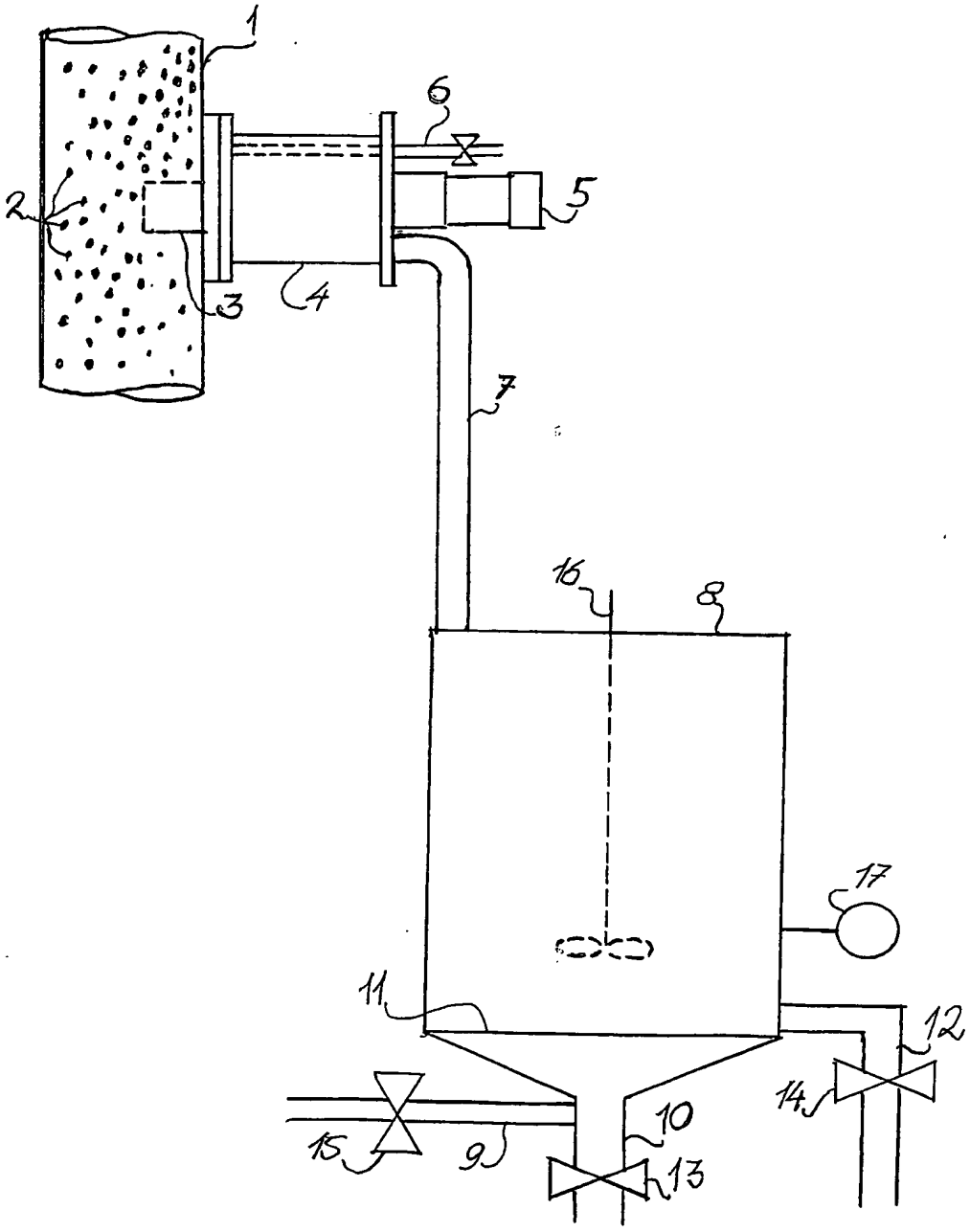
11. A system of apparatus according to Claims 9-10, **characterized** in that the dilution space (8) includes means (16) for additional agitation of the resultant pulp suspension.

12. A system of apparatus according to Claims 9-11, **characterized** in that a pulp consistency measuring device (17) is connected to the dilution space.

13. A system of apparatus according to Claims 9-12, **characterized** in that the means for de-watering the pulp fibre suspension consists of a wire cloth (11) of given mesh size fastened to the walls of the dilution space (8) or resting on a perforated substrate anchored to said dilution space (8).

14. A system of apparatus according to Claims 9-13, **characterized** in that said conduits include openable and closeable valves (13, 14, 15), the settings of which make possible the various method steps.

Fig.1



INTERNATIONAL SEARCH REPORT

International application No.
PCT/SE 02/02189

A. CLASSIFICATION OF SUBJECT MATTER

IPC7: G01N 1/38, G01N 1/20
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC7: G01N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-INTERNAL, WPI DATA, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	Patent Abstracts of Japan, abstract of JP 64-14386 A (ISHIKAWAJIMA HARIMA HEAVY IND CO LTD), 18 JAnuari 1989 (18.01.89), abstract --	1-14
A	US 3747411 A (MCDERMOTT, W.F. ET AL), 24 July 1973 (24.07.73), see the whole document --	1-14
A	US 3942388 A (RATHNOW, E. ET AL), 9 March 1976 (09.03.76), see the whole document --	1-14
A	US 5625157 A (PIIRAINEN, E. ET AL), 29 April 1997 (29.04.97), see the whole document --	1-14

[X] Further documents are listed in the continuation of Box C. [X] See patent family annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
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INTERNATIONAL SEARCH REPORT

International application No.
PCT/SE 02/02189

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

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INTERNATIONAL SEARCH REPORT
Information on patent family members

30/12/02

International application No.
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